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High order reflectivity of graphite (HOPG) crystals for x ray energies up to 22 keV

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We used Kr $K\alpha$ (12.6 keV) and Ag $K\alpha$ (22.1 keV) x-rays, produced by petawatt class laser pulses interacting with a Kr gas jet and a silver foil, to measure the integrated crystal reflectivity of flat Highly Oriented Pyrolytic Graphite (HOPG) up to fifth order. The reflectivity in fourth order is lower by a factor of 50 when compared to first order diffraction. In second order the integrated reflectivity decreases from 1.3 mrad at 12.6 keV to 0.5 mrad at 22.1 keV. The current study indicates that HOPG crystals are suitable for measuring scattering signals from high energy x ray sources ($E \geq 20$ keV). These energies are required to penetrate through the high density plasma conditions encountered in inertial confinement fusion capsule implosions on the National Ignition Facility.

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I. INTRODUCTION

Highly Oriented Pyrolytic Graphite (HOPG) crystals are of particular interest for x-ray diagnostics of hot dense plasmas. Their unique crystal plane structure enables them to be highly efficient x-ray diffraction instruments. These type of crystals, for example, have been successfully used in novel x-ray scattering experiments on warm dense matter [1–3] for x-ray energies of 3 to 9 keV. On the other hand, photon energies > 10 keV are required for the probing of super-dense states of matter, as encountered in inertial confinement fusion experiments [4]. The aim of this work is to characterize the reflectivity of HOPG in this energy range, i.e. at 12.6 keV and 22.1 keV. Since the Bragg angle decreases when going to higher photon energies, it is necessary to use the crystals in higher diffraction orders. We have characterized the performance of HOPG in up to fifth order to ascertain their effectiveness for use in x-ray scattering experiments, especially when the detection of weak signals requires high reflectivity and good spectral resolution.

II. EXPERIMENTAL SETUP

We used the petawatt-class Titan laser facility at the Lawrence Livermore National Laboratory [citation needed] to produce $K\alpha$ x-rays at 12.6 keV (Kr $K\alpha$) and 22.1 keV (Ag $K\alpha$). In these experiments, the Titan short-pulse beam delivered a pulse energy of up to 180 J at 1054 nm in 0.7 ps. Focusing is achieved with an f/3 off-axis parabola to a $1/e^2$ spot diameter of 15 μm , yielding an intensity on target above 10^{20} W/cm². To generate Kr $K\alpha$ x rays, the laser was focused onto a super sonic Kr gas jet [5]. We aligned the laser focus close to the gas jet nozzle and near the front edge of the gas jet in order to maximize conversion efficiency [6]. The backing pressure was kept at 90 bar to ensure the generation of large clusters and thus efficient laser absorption [7]. The neutral density of the gas jet was measured off line us-

ing a Lloyd's mirror interferometer to be $8.7 \times 10^{19} \text{ cm}^{-3} \pm 10\%$ on axis at the nozzle exit. For generating Ag $K\alpha$ x rays the laser pulses were focused on a 10 μm silver foil.

The x-ray conversion efficiencies were measured on each shot using a single hit counting CCD [8, 9] to be $> 10^{-5}$ [5] and $> 10^{-4}$ [8] into the full solid angle for Kr $K\alpha$ and Ag $K\alpha$, respectively. This technique uses a CCD camera placed far enough away from the x-ray source that a series of single photon events are recorded on the detector. Single event histogram binning [9] of the image yields an absolute number of photon events at discrete energies. In our setup, the single hit CCD observed the front side of the gas jet from distance of 5 m.

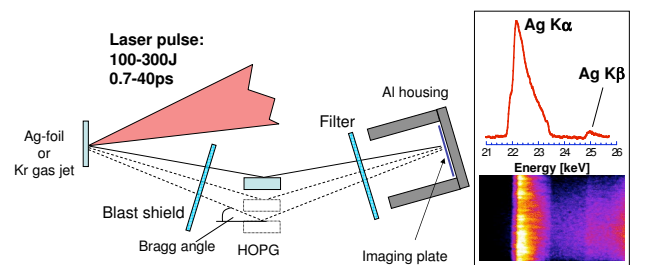


FIG. 1: Schematics of the experimental setup. For the Kr $K\alpha$ setup a focal length (distance from source to HOPG) of 230 mm was used. The inset shows a sample raw image for Ag $K\alpha$. As a reference for absolute reflectivity measurements a single hit CCD camera and an additional HOPG spectrometer in fixed geometry (second order) were operated (not shown).

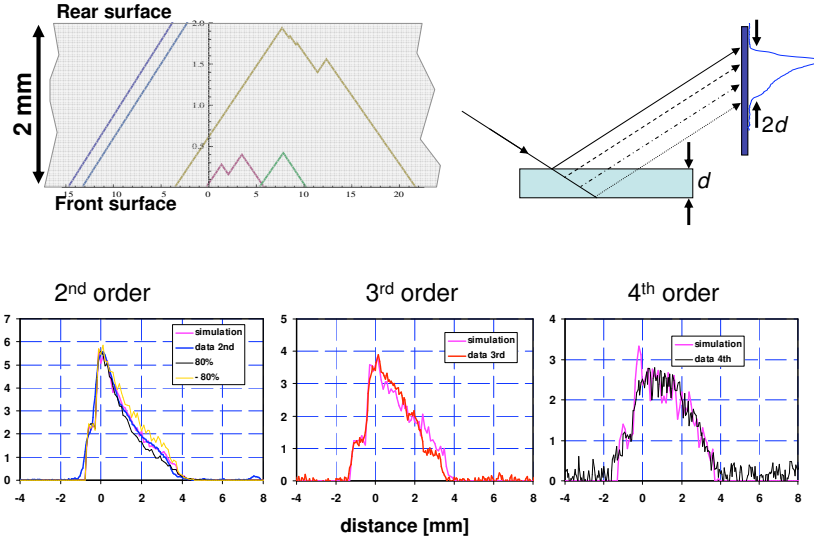


FIG. 2: The lineshape at 22.1 keV is dominated by depth broadening which can be reproduced using Monte Carlo simulations. This method can serve as an independent means to determine reflectivity changes between different HOPG orders. [will improve statistics of fit function]

High resolution spectra of the $K\alpha$ line emission were recorded using two HOPG spectrometers. For diffraction we used $25.4 \text{ mm} \times 25.4 \text{ mm} \times 2 \text{ mm}$ ZYB grade HOPG crystals (Advanced Ceramics) with a mosaic spread of $\gamma = 0.8$ degree. The separation between the mosaic crystal planes is $d = 0.3354 \text{ nm}$. While one spectrometer was operated in second order for all the shots to serve as an additional source monitor, the primary instrument was used to measure the integrated HOPG reflectivities in various diffraction orders, see Fig. 1 for the schematics of the setup. For detection we used imaging plates (Fuji BAS-SR), which previously were absolutely calibrated at 5.9 keV and 22 keV [10, 11]. In between the imaging plate sensitivity can be extrapolated by the absorption properties of the plates. In the gas jet setup the spectrometer was filtered using $25 \mu\text{m}$ Mylar as blast shield, and $25 \mu\text{m}$ Beryllium plus $12 \mu\text{m}$ Aluminum in front of the imaging plates. Differential filtering using $25 \mu\text{m}$ Pb was used to determine contribution from different diffraction orders. For Ag $K\alpha$ $25 \mu\text{m}$ and $400 \mu\text{m}$ Aluminum were used as blast shield and imaging plate filter, respectively.

III. RESULTS AND DISCUSSION

For mosaic focusing, the Bragg relationship must be satisfied: $n\lambda = 2d \sin \theta_B$, where n is the diffraction or-

der, λ is the x-ray wavelength, and θ_B is the Bragg angle. For Ag $K\alpha$ θ_B is very small for low diffraction orders, which makes it challenging to align, and, in particular for first order, requires very large crystals to cover the necessary bandwidth. At 12.6 keV we measured all diffraction orders from one to five. In fifth diffraction order, the signal was below the detection threshold. At 22.1 keV the reflectivity was measured from second to fourth order. Fig. 2 shows the Ag $K\alpha$ line shapes in second to fourth order. In general the measured line width is determined by a variety of factors, i.e. the natural line width, depth broadening, surface roughness, mosaicity of the crystal, and the source size, which was discussed, e.g., in Ref. [12]. At higher energies in general, and at 22.1 keV in particular the line width is dominated by depth broadening which appears on the high energy side

Kr $K\alpha$ (12.6 keV)	n	θ_B	integrated reflectivity [mrad]	Ag $K\alpha$ (22.1 keV)	n	θ_B	integrated reflectivity [mrad]
	1	8.4	3.7		1	4.8	
	2	17.0	1.3		2	9.7	0.47
	3	26.1	0.21		3	14.6	0.12
	4	35.9	0.077		4	19.6	0.034
	5	47.1	<0.02		5	24.8	

TABLE I: Bragg angles and measured integrated reflectivities

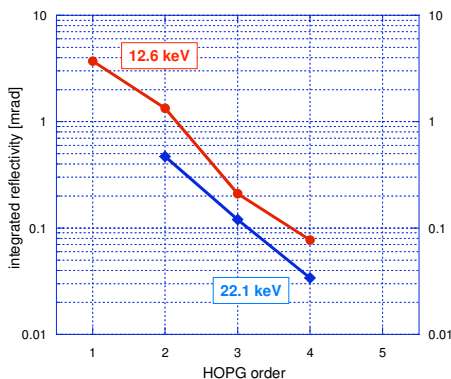


FIG. 3: Measured integral HOPG reflectivities at 12.6 keV (Kr $K\alpha$) and 22.1 keV (Ag $K\alpha$) up to 4th order. For 12.6 keV the setup in 5th order was tested, but the signal was below the detection threshold.

of the main line, see Fig. 2. Nevertheless, the instrument resolution is good enough to resolve Ag $K\alpha_1$ and Ag $K\alpha_2$. Since the attenuation length of carbon at 22.1 keV is 15 mm, the depth broadening is limited by the thickness of the crystal. Since the energy resolution increases to higher diffraction order, the spectral line width decreases accordingly. Thus decreasing the thickness of the crystal is a possibility to enhance the energy resolution of the instrument, though sacrificing reflectivity.

We performed Monte Carlo simulations to model the shape of the measured Ag $K\alpha$ lines. Within this simplified model the line shape depends on the mosaicity of the crystal, the width of the crystal, the Bragg angle, the attenuation length in graphite, as well as the reflectivity R per unit length, which is adjusted to fit the synthetic

line shape to the measured profile. For the second order signal in Fig. 2 the variation of the fit function with respect to R is illustrated. The lowest diffraction order is dominated by single reflections yielding an exponential decay in the high energy side. For higher diffraction orders the probability of multiple reflections within the crystal increases, significantly changing the shape of the measured line. The reflectivities $R^{(n)}$ extracted in this way from the simulation show the same dependence on the diffraction order n as the measured integrated reflectivity. Hence at high x ray energies where the line width is dominated by depth broadening the measurement of the line shape is a viable way of determining crystal reflectivities.

The measured integrated reflectivities are shown in Fig. 3. In general, the signal strength decreases towards higher diffraction orders by two orders of magnitude. Even though the absolute values differ the slope is very similar at 12.6 keV and 22.1 keV. The highest reflectivity is observed at 12.6 keV in first order with 3.7 mrad. This value agrees with previous measurements at ~ 5 keV and ~ 8 keV [12]. At 22.1 keV the reflectivity is measured to be lower by a factor of 2 to 3 compared to 12.6 keV. Nevertheless, the integrated reflectivity is still up to about a factor of 5 times higher than for commonly used less mosaic crystals such as LiF or PET [13] making them suited for the detection of weak signals, as required in x-ray Thomson/Compton scattering experiments to probe dense and warm states of matter.

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- [1] S. H. Glenzer, G. Gregori, R. W. Lee, F. J. Rogers, S. W. Pollaine, and O. L. Landen. Demonstration of spectrally resolved x-ray scattering in dense plasmas. *Phys. Rev. Lett.*, 90(17), 2003.
 - [2] G. Gregori, S. H. Glenzer, H. K. Chung, D. H. Froula, R. W. Lee, N. B. Meezan, J. D. Moody, C. Niemann, O. L. Landen, B. Holst, R. Redmer, S. P. Regan, and H. Sawada. Measurement of carbon ionization balance in high-temperature plasma mixtures by temporally resolved x-ray scattering. *J. Quant. Spectr. Rad. Trans.*, 99(1-3):225, 2006.
 - [3] S. H. Glenzer, O. L. Landen, P. Neumayer, R. W. Lee, K. Widmann, S. W. Pollaine, R. J. Wallace, G. Gregori, A. Höll, T. Bornath, R. Thiele, V. Schwarz, W. D. Kraeft, and R. Redmer. Observations of plasmons in warm dense matter. *Phys. Rev. Lett.*, 98(6), 2007.
 - [4] J. D. Lindl, P. Amendt, R. L. Berger, S. G. Glendinning, S. H. Glenzer, S. W. Haan, R. L. Kauffman, O. L. Landen, and L. J. Suter. The physics basis for ignition using indirect-drive targets on the national ignition facility. *Phys. Plasmas*, 11(2):339, 2004.
 - [5] N. L. Kugland, C. G. Constantin, C. Niemann, P. Neumayer, H.-K. Chung, T. Döppner, A. Kemp, S. H. Glenzer, and F. Girard. 12.6 keV Kr $K\alpha$ x-ray source for high energy density physics experiments. *submitted to Rev. Sci. Instrum.*, 2008.
 - [6] N. L. Kugland et al. *submitted to Appl. Phys. Lett.*, 2008.
 - [7] T. Ditmire, R. A. Smith, J. W. G. Tisch, and M. H. R. Hutchinson. High Intensity Laser Absorption by Gases

- of Atomic Clusters. *Phys. Rev. Lett.*, 78:3121–4, 1997.
- [8] H. S. Park, D. M. Chambers, H. K. Chung, R. J. Clarke, R. Eagleton, E. Giraldez, T. Goldsack, R. Heathcote, N. Izumi, M. H. Key, J. A. King, J. A. Koch, O. L. Landen, A. Nikroo, P. K. Patel, D. F. Price, B. A. Remington, H. F. Robey, R. A. Snavely, D. A. Steinman, R. B. Stephens, C. Stoeckl, M. Storm, M. Tabak, W. Theobald, R. P. J. Town, J. E. Wickersham, and B. B. Zhang. High-energy k alpha radiography using high-intensity, short-pulse lasers. *Phys. Plasmas*, 13(5), 2006.
- [9] B.R. Maddox et al. *submitted to Rev. Sci. Instrum.*, 2008.
- [10] N. Izumi, R. Snavely, G. Gregori, J. A. Koch, H. S. Park, and B. A. Remington. Application of imaging plates to x-ray imaging and spectroscopy in laser plasma experiments (invited). *Review of Scientific Instruments*, 77(10), 2006.
- [11] N. Izumi. *private communication*.
- [12] A. Pak, G. Gregori, J. Knight, K. Campbell, D. Price, B. Hammel, O. L. Landen, and S. H. Glenzer. X-ray line measurements with high efficiency bragg crystals. *Review of Scientific Instruments*, 75(10):3747, 2004.
- [13] F. J. Marshall and J. A. Oertel. Framed monochromatic x-ray microscope for icf (invited). *Review of Scientific Instruments*, 68(1):735, 1997.